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Patentanmeldung Nr. Patent application No. Demande de brevet n°

03103838.3



Der Präsident des Europäischen Patentamts;  
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets  
p.o.

R C van Dijk



Anmeldung Nr:  
Application no.: 03103838.3  
Demande no:

Anmeldetag:  
Date of filing: 16.10.03 /  
Date de dépôt:

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Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:  
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.  
If no title is shown please refer to the description.  
Si aucun titre n'est indiqué se référer à la description.)

Process for the continuous production of emulsions PROCESS FOR PRODUCING SAME

In Anspruch genommene Priorität(en) / Priority(ies) claimed /Priorité(s)  
revendiquée(s)  
Staat/Tag/Aktenzeichen/State/Date/File no./Pays/Date/Numéro de dépôt:

Internationale Patentklassifikation/International Patent Classification/  
Classification internationale des brevets:

B01F/

Am Anmeldetag benannte Vertragstaaten/Contracting states designated at date of  
filing/Etats contractants désignées lors du dépôt:

AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LU MC NL  
PT RO SE SI SK TR LI

**TITLE:****Process for the continuous production of emulsions**

The present invention relates to a process for the continuous preparation of emulsions with uniform size distribution of the droplets forming the dispersed phase of the emulsion.

In particular, the present invention relates to a process for the continuous preparation of emulsions containing, as dispersed phase, droplets of a molten adduct of magnesium halide-Lewis base, in which emulsion the size distribution of the droplets is particularly narrow.

The most immediate and specific application of the present invention is the production in continuous of emulsions which, after a cooling step, allows to obtain solid particles of a magnesium dihalide-Lewis base adduct having a more uniform size distribution. The obtained solid particles, in view of their narrow particle size distribution, can be advantageously used as a support in the preparation of supported catalysts for the polymerization of olefins.

It is known that industrial mixers and emulsifiers are used to blend various materials, such as adhesives, coatings, cosmetics, foods, plastics, etc. Generally, high speed and hydraulic shearing forces are created inside the industrial mixers by rotating a rotor relative to a stator: the fluids are fed axially into the rotor-stator assembly and dispersed radially outward from the rotor-stator assembly. When two immiscible liquids are subjected to high speed and hydraulic shearing forces, one liquid can disperse in the form of droplets (dispersed phase) inside the second one (continuous phase) causing the formation of an emulsion. The average size of the droplets in the dispersed phase is directly correlated to the intensity of the energy supplied by the rotor. Generally, the higher is the stirring energy, the smaller is the average size of the droplets obtained as dispersed phase of the emulsion. Another important parameter, which greatly influences the final size of the obtained

droplets, is the degree of coalescence during the emulsifying process. In such processes the droplets of the emulsion are substantially in dynamic equilibrium between the breakup involved by the shearing action and the coalescence phenomena tending to aggregate the droplets. The coalescence of the droplets causes an increase of the final average size of the droplets obtained at the outlet of the process, thus increasing considerably the fraction of the coarse particles. Such coalescence phenomena are contrasted by the shearing action caused by the movement of the rotor. In the mixing zones close to the rotor the shearing action is intense and, consequently, droplets having a small size are formed. Moving away from the rotor, the shearing action decreases and the coalescence phenomena increase, so causing an increase of the size of the droplets. Bigger droplets are formed in the zones of mixing at some distance from the rotor. As a consequence, the obtained emulsion shows a non-uniform size distribution of the droplets.

USP 5,622,650 discloses a process for emulsifying a hydrophobic fluid and a hydrophilic fluid in order to obtain emulsions, particularly oil-in-water emulsions. Said oil-in water emulsions are employed in the preparation of microcapsules for use in the manufacture of carbonless copy papers. Such emulsifying process makes use of a milling machine comprising a mill body having a substantially cylindrical cavity therein. The milling machine further comprises a plurality of annular, concentric rows of stator projections and a plurality of annular, concentric rows of rotor projections, wherein the annular rows of stator projections are concentrically adjacent to, and are nested or intermeshed within, the annular rows of rotor projections. The fluids entering the mill pass through shear zones of increasing intensity, before reaching the outer periphery of the rotor member. The fluids flow radially through a first milling space and axially through a second milling space, within the close peripheral clearance between the knurled outer circumference of the rotor (FE6138-EP)

member and the inside wall of the cavity, before the fluids reach the mill outlet. The most important feature of the milling machine of USP 5,622,650 is the use of a single pass continuous feed process to produce emulsions having a more uniform particle size. Said single pass process is specific for the preparation of microcapsules for use in the manufacture of carbonless copy papers and is not industrially transferable to the preparation of emulsions which, once quenched, can give solid particles to be used for preparing supported catalysts.

USP 5,643,506 describes a process for preparing microcapsules having a substantially uniform size distribution, which comprises continuously forming an oil-in-water emulsion, in which the hydrophobic phase is about 45% to 95% by volume and the hydrophilic phase is about 5 to 55% by volume and where the particle size of the emulsion is controlled by adjusting the oil to water ratio within the aforesaid ranges. This method of controlling the final particle size distribution of the emulsion is not industrially transferable to the preparation of solid catalytic components. In such a preparation it is preferable to avoid the regulation of the particle size distribution by acting on the ratio between the dispersed phase and the continuous phase. A reduction of the amount of the dispersed phase causes a consequent reduction of the generated solid particles bringing to a drastic decrease of the process productivity. Furthermore, the process of USP 5,643,506 discloses the addition of particular chemical compounds (isocyanates) to the hydrophobic phase and to the hydrophilic phase. The addition of such chemical compounds causes a modification of the physical and chemical characteristics of the obtained emulsion. This method is not transferable to the preparation of solid catalytic components, since the above chemical compounds can cause a drastic reduction of the activity of the final catalyst.

USP 5,632,596 relates to a rotor-stator assembly for producing sub-micron droplet size (FE6138-EP)

liquid-liquid emulsions, usable in industrial mixers and emulsifiers for a variety of mixing operations, e.g. mixing, emulsifying, homogenizing, dissolving, dispersing, blending, etc.

The particular configuration of the rotor-stator assembly of USP 5,632,596 causes an increase of the shearing action of the apparatus on the treated mixture. Said rotor-stator assembly includes a rotor and a stator, each of which has a plurality of teeth wherein the height of the teeth is less than approximately one twentieth the diameter of the rotor-stator.

USP 6,000,840 relates to rotor-stator assemblies suitable for industrial mixers and emulsifiers, the mechanical configuration of which represents an alternative to that described in USP 5,632,596. The particular configuration of the rotor-stator assemblies of USP 6,000,840 allows to transmit increased and/or additional high speed shearing forces and increased pressures on the treated mixture. The stator of USP 6,000,840 comprises a plurality of openings, said plurality of openings being arranged in pairs in a generally V-shaped pattern. The rotor of the above assembly comprises a plurality of blades and means for supporting said blades for rotation about an axis of rotation, wherein at least one of said blades comprises a surface disposed at an oblique angle relative to said axis of rotation.

Both the rotor-stator assemblies of USP 5,632,596 and 6,000,840 are aimed to improve the mixing effect in a mixing stage and do not take in any consideration the coalescence phenomena occurring downstream a mixing stage. As above explained, the coalescence phenomena are particularly relevant in causing an increase of the size of the droplets and, as a consequence, the obtained emulsions are characterized by a non-uniform size distribution of the droplets. Furthermore, the high degree of coalescence in the conventional prior art processes is an undesirable effect: in fact, when the coalescence phenomena are out of control, they cause the formation of emulsions having a non-uniform, broader size distribution of the droplets in the dispersed phase. Such drawback must be minimized if the (FE6138-EP)

obtained emulsion has to be used in the preparation of supported catalysts for the polymerization of olefins. In fact, a size distribution of the support as much as possible narrow is highly desirable in this specific application.

In view of the above, it would be desirable to have available a process for continuously preparing emulsions wherein the coalescence phenomena are considerably minimized and wherein the efficiency of the process, in term of energy transferred to the fluids, is improved.

It has been now found a process for preparing emulsions of two immiscible liquids, in which process the coalescence phenomena are minimized, thus obtaining emulsions characterized by a narrow size distribution of the droplets in the dispersed phase.

It is therefore an object of the present invention a multistage process for the continuous production of an emulsion comprising subjecting at least two immiscible liquids to a sequence of at least two mixing stages carried out in at least two successive stator-rotor devices, wherein a peripheral outlet from a first stator-rotor device is connected to an axial inlet in the successive stator-rotor device by means of a duct in which the Reynold number  $Re_T$  inside said duct is higher than 5000, and the peripheral velocity of each rotor of said stator-rotor devices ranges from 5 to 60 m/s.

The peripheral velocity is defined as the product of the angular rotation velocity and the radius of the rotor.

The Reynolds number relative to the movement of a fluid inside a tube ( $Re_T$ ) is defined by the formula  $Re = D \cdot v \cdot d / \eta$  in which  $D$  is the diameter of the tube,  $v$  is the linear velocity of the fluid,  $d$  is its density and  $\eta$  is the dynamic viscosity.

According to the process of the present invention, the mixing of the liquids is carried out in one or more rotor-stator devices, each mixing stage being connected to the successive one (FE6138-EP)

by means of one or more ducts connecting a peripheral outlet from a stator-rotor device to an axial inlet in the successive stator-rotor device.

Throughout the present specification, the term "peripheral outlet" means that the outlet is placed in proximity of the outer circumference of the rotor, the term "axial inlet" means that the inlet is placed in the central portion of the stator-rotor device, that is to say in proximity of the rotor axis.

The process of the invention will be now described with reference to the preparation of an emulsion in which the dispersed phase comprises a molten adduct of magnesium dihalide-Lewis base, while the continuous phase of the emulsion is a liquid which is inert and immiscible with respect to said adduct of Mg dihalide-Lewis base. Such liquid is preferably an organic liquid selected from aliphatic and aromatic hydrocarbons, silicone oils, liquid polymers or mixtures of said compounds. Paraffin oils and silicone oils having a viscosity between 30 cSt and 300 cSt at room temperature are particularly preferred.

As regards the feeding ratio of the above components, the molten adduct of magnesium dihalide-Lewis base is fed to the first mixing stage with a weight ratio of generally less than 0.5, preferably of less than 0.3, with respect to the other liquid component.

It has been unexpectedly found that when the above indicated values of peripheral velocity and  $Re_T$  are imposed, a minimization of the coalescence phenomena throughout the process of emulsification is obtained. In particular, the best results in term of minimization of the coalescence phenomena and improvement of droplets size distribution are obtained when the following process parameters are satisfied:

a Reynold number  $Re_T$  higher than 8000 inside each duct,

a peripheral velocity of the rotor in the range from 20 to 60 m/s,

a residence time of less than 1 second in each mixing stage.

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The residence time in a mixing stage is defined as the ratio between the volume occupied by the liquid in the mixing stage and the volumetric rate at the outlet from the mixing stage. According to the process of the present invention, the above immiscible liquids are fed to a first mixing stage carried out in a first stator-rotor device. The inlet of the two liquids is placed in proximity of the rotor axis (axial inlet), so that the entering liquids are forced by the rotation of the rotor to flow from the rotor axis towards the peripheral rim of the rotor. In this way the above liquid components run at a great velocity inside the limited space comprised between the stator and the rotor surface. Due to the strong stirring and shearing action involved by the rotation of the rotor, an emulsion of the above liquids is formed. Due to the mutual immiscibility, the molten adduct of magnesium dihalide-Lewis base is caused to be dispersed in the form of little droplets in the liquid fed as the continuous phase.

When the obtained emulsion reaches the peripheral region of the first mixing stage, it is forced to enter a duct connecting a peripheral outlet from the first stator-rotor device to an inlet placed in the central portion of a successive stator-rotor device. The transfer of the emulsion along the connecting duct is achieved by exploiting the energy provided by the rotor disk. Once introduced inside the second mixing stage, the emulsion is forced by the rotation of a second rotor disk to flow at a great velocity from the rotor axis towards the peripheral rim of the rotor. The same effects, as in the first stage, are obtained in the second mixing stage in term of shearing and stirring. When the emulsion reaches the peripheral area of the second mixing stage, it is forced to enter another duct connecting a peripheral outlet from the second stator-rotor device to an axial inlet in the successive stator-rotor device.

In view of the intense shearing action provided by each rotor disk, a turbulent flow is established when the emulsion runs through the ducts connecting the mixing stages each (FE6138-EP)

other. As above indicated, the Reynolds number  $Re_T$  relative to the movement of the emulsion inside the ducts is generally higher than 5000. It should be noted that values of  $Re$  below 2000 correspond to laminar flow, while values of  $Re$  above 4000 correspond to a turbulent flow. The zone between 2000 and 4000 is the so-called transition zone. The connecting ducts have preferably a spiral shape: this improves the efficiency of the process in term of reduction of the coalescence phenomena between a mixing stage and the successive one.

The stages successive to the first mixing stage have the function of improving the efficiency of the process of emulsification by increasing the amount of energy provided to the fluids. The obtained emulsion is then discharged from the last mixing stage through a peripheral outlet placed in correspondence of the last stator.

In the emulsifying process of the present invention the mixing energy is provided to the fluids more uniformly with respect to the prior art processes. The multistage process of the present invention is particularly innovative, because it allows to use more stages of mixing overcoming the prior art problems caused by the coalescence phenomena between one stage and the successive one. In fact, the size distribution of the droplets obtained at the outlet of each mixing stage is maintained substantially unchanged at the inlet of the successive mixing stage by effect of the specific connection between the stator-rotor devices. By virtue of the high turbulence intensity ( $Re_T > 5000$ ) provided inside each connecting duct, the process of the present invention allows to minimize the coalescence phenomena between each emulsifying stage. As a consequence, the results obtained in the first mixing stage in term of size distribution of the droplets can be transferred without any substantial modification to the second mixing stage where the droplets size distribution is further improved. This is the most relevant advantage provided by the present invention. In (FE6138-EP)

fact, the minimization of the coalescence phenomena allows the preparation of an emulsion characterized by a more uniform distribution of the droplets.

The same amount of energy may be transmitted to the liquid components in each mixing stage or, alternatively, an increasing amount of energy can be transmitted passing from a mixing stage to the successive one. In fact, it is possible to increase the energy provided in a rotor-stator device by properly selecting the peripheral velocity of the rotor or, in alternative, by selecting the tolerance between the lateral surface of the rotor and the walls of stator. Also by the use of rotor disks having a different thickness, it is possible to modify said tolerance, so increasing or decreasing the energy provided in a mixing stage. According to a preferred embodiment, the process of the invention comprises a sequence of three mixing stages.

As regards the molten adduct of magnesium dihalide-Lewis base, the use of  $\text{MgCl}_2$  as a magnesium dihalide is preferred. The Lewis base forming the adduct with the Mg dihalide is preferably selected from the group consisting of amines, alcohols, esters, phenols, ethers, polyethers and aromatic or aliphatic (poly)carboxylic acids. Among them, particularly preferred are the alcohols of formula  $\text{ROH}$  in which R is an alkyl group containing from 1 to 10 carbon atoms.

Especially preferred adducts are those of formula  $\text{MgCl}_2 \cdot m\text{ROH} \cdot n\text{H}_2\text{O}$  in which m ranges from 0.1 to 6, n ranges from 0 to 0.7 and R has the meaning given above. Among them particularly preferred adducts are those in which m ranges from 2 to 4, n ranges from 0 to 0.4 and R is ethyl.

It is another object of the present invention an apparatus for the continuous production of an emulsion comprising a plurality of at least two stator-rotor devices, each stator except the last being connected with the successive stator by a duct extending from a peripheral outlet (FE6138-EP)

in the first stator to an axial inlet in the successive stator. Preferably, the initial portion of said ducts is oriented in a direction substantially tangential to the circumference of each rotor and the terminal portion of said ducts is oriented in a direction substantially parallel to the rotation axis of each rotor.

The efficiency of the above apparatus in term of reduction of the coalescence phenomena is improved when the ducts conveying the emulsion from a rotor-stator device to the successive one are shaped as a spiral.

Each rotor is generally perforated by one or more small holes allowing the liquids of the emulsion to pass from one side to the other side of the rotor. In such a way, both the spaces comprised between the lateral surfaces of the rotor and the stator are exploited and subjected to the shearing action of the rotor. Optionally, the circumference of each rotor may be slightly notched in order to prevent coalescence phenomena in the layer of fluid more adjacent to the stator, wherein the coalescence phenomena are more likely to occur.

The axial tolerance between each rotor and the corresponding stator is generally comprised in the range from 0,1 to 2,0 mm, preferably from 0,2 to 1,2 mm. The axial tolerance may be defined as the distance comprised between the lateral surface of a rotor and the lateral surface of the corresponding stator.

The radial tolerance, intended as the radial distance between the circumference of each rotor and the corresponding stator, is generally comprised between 0,2 mm and 5,0 mm, preferably between 0,5 and 2,0 mm.

The above values of axial and radial tolerances are easily selected by a simple substitution of the dimensions of the rotor disks. This is an advantage providing simplicity to the construction and maintenance of the apparatus.

The apparatus of the invention comprises at least two rotor-stator devices. In a preferred (FE6138-EP)

embodiment of the invention, the apparatus comprises three rotor-stator devices and the axial tolerance between the rotor disk and the corresponding stator is decreasing from a mixing stage to the successive one.

A scheme of the process and apparatus of the present invention is given with reference to the accompanying drawings, which are given for illustrative purpose not limiting the scope of the invention.

Fig. 1 is a representation of the process and apparatus of the invention, where a sequence of three mixing stages is shown.

Fig. 2 is a front view of the apparatus shown in Fig. 1.

With reference to Fig.1, a magnesium dihalide-Lewis base adduct 1 and a liquid 2 immiscible with said adduct are fed to a first mixing stage carried out in a first stator-rotor device. An axial inlet 3 is provided in the stator 4, so that the entering liquids are forced by the rotation of the rotor 5 to flow from the rotor axis 6 towards the peripheral rim of the rotor. In this way the liquid components run at a great velocity inside the restricted space comprised between the stator 4 and the lateral surface of rotor 5.

Due to the strong stirring and shearing action involved by the rotation of the rotor 5, an emulsion of the above liquids is formed. When the emulsion reaches the peripheral region of the first mixing stage, it is forced to enter a duct 7 connecting a peripheral outlet 8 from the first stator-rotor device to an axial inlet 9 placed in the central portion of the successive stator-rotor device.

Once introduced inside the second rotor-stator device, the emulsion is forced by the rotation of a second rotor 10 to flow at a great velocity from the rotor axis towards the peripheral rim of the rotor 10. When the emulsion reaches the peripheral area of the second mixing stage, it is forced to enter another duct 11 connecting a peripheral outlet 12 from the second

stator-rotor device to an axial inlet 13 in the successive stator-rotor device. The emulsion is forced by the rotation of the rotor 14 to flow from the rotor axis to the peripheral rim of the rotor 14 and then it is discharged from the third mixing stage through a duct 15.

With reference to Fig. 2, a front view of the first rotor-stator device of Fig. 1 is shown. An axial inlet 3 of the liquids is provided around the rotor axis 6 and a tangential and peripheral outlet 8 from the rotor-stator device is provided.

The emulsions obtained by the process of the present invention have shown excellent properties as regards the size distribution of the droplets and the morphological properties of the products obtained by quenching the droplets.

In fact, the most important application of the process of the invention is the continuous preparation of emulsions which, once quenched under suitable conditions, give rise to the formation of solid particles having a more uniform size distribution. A preferred method for cooling the emulsion is described in the patent application WO 02/051544. The solidification of the magnesium dihalide-Lewis base adduct, contained in the emulsion in form of droplets, gives solid particles, which, in view of their narrow particle size distribution, can be advantageously used as a support in the preparation of supported catalysts for the polymerization of olefins.

In fact, in the preparation of supported catalysts for the polymerization of olefins, it is highly desirable to obtain a size distribution of the support particles as much as possible narrow in the ranges of average values requested in this specific application.

The support particles obtained by quenching the above obtained emulsion are characterized by an average size in the range of from 4 to 120  $\mu\text{m}$ .

The particle size distribution can be valued according to the  $\text{SPAN} = \frac{P90 - P10}{P50}$  where, in

a particle size distribution curve, P90 is the value of the diameter such that 90% of the total particles have a diameter lower than that value, P10 is the value of the diameter such that 10% of the total particles have a diameter lower than that value and P50 is the value of the diameter such that 50% of the total particles have a diameter lower than that value. It is clear that a low SPAN value implies a narrow particle size distribution.

In the particular case of an adduct of formula  $\text{MgCl}_2 \cdot m\text{ROH}$  wherein  $m$  ranges from 0.1 to 6, and R is an alkyl group containing from 1 to 10 carbon atoms, the process of the invention allows to obtain a droplet size distribution according to the formula  $\frac{P90 - P10}{P50}$

of less than 1.2 and preferably of less than 1.0.

The process provided by the present invention is extremely advantageous since it allows to select the average value of the particle size distribution in a wide range (4-120  $\mu\text{m}$ ), while maintaining a narrow size distribution of the particles ( $\text{SPAN} < 1.2$ ). These results are shown in the working examples of the present patent application and are obtained without the addition to the emulsion of chemical compounds, surfactants and other additives. As said, these chemical compounds are not compatible with the preparation of solid catalytic components, since they can cause a drastic reduction of the activity of the final catalyst.

The obtainment of solid catalytic components having a distribution curve as much as possible narrow reduces also the formation of fines during the polymerization of  $\alpha$ -olefins. This is particularly advantageous because in a gas-phase polymerization the fines are responsible of the formation of electrostatic charges inside the polymerization reactor and cause the fouling of the reactor walls. Furthermore, the fines are easily entrained by gaseous stream recycled to the bottom of the polymerization bed for cooling the reactor and this recycle of fines is not desired.

The following examples have to be considered representative and non-limiting the scope of the invention.

### EXAMPLES

In examples 1-3 the process of the invention is carried out according to the embodiment shown in Fig. 1 where three mixing stages are used.

The comparative example 4 is carried out in a single-stage apparatus comprising only the first rotor-stator device of Fig. 1 (stator 4 and rotor 5) from which the obtained emulsion is discharged without passing to the successive rotor-stator device.

The below mentioned parameters can be defined as follows:

$Re_T = D \cdot v \cdot d / \eta$ , wherein  $D$  is the tube diameter,  $v$  is the linear velocity of the fluid,  $d$  is the fluid density and  $\eta$  is the fluid dynamic viscosity;

Peripheral velocity = product of the rotor angular velocity and radius of the rotor;

$SPAN = \frac{P90 - P10}{P50}$  wherein  $P90$  is the value of the diameter such that 90% of the total

particles have a diameter lower than that value,  $P10$  is the value of the diameter such that 10% of the total particles have a diameter lower than that value and  $P50$  is the value of the diameter such that 50% of the total particles have a diameter lower than that value.

#### Example 1

The following liquid components are fed at 125°C to the axial inlet of the first rotor-stator device:

- a mineral oil (dynamic viscosity at 40°C of 52 cPoise and density of 865 kg/m<sup>3</sup>) as the continuous phase of the emulsion;
- a molten adduct of formula  $MgCl_2 \cdot 2,7C_2H_5OH$  as the dispersed phase of the emulsion.

The feeding ratio (by weight) between the molten adduct and the mineral oil is 0,13.

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The operative conditions are such that the residence time inside each rotor-stator device is maintained at a value of 0,1 seconds, while the peripheral velocity of the rotor disks is 7,0 m/s. The  $Re_T$  inside the ducts connecting the mixing stages is of about 8200.

An emulsion comprising the molten adduct as the dispersed phase and the above mineral oil as the continuous phase is discharged from the outlet of the third mixing stage. The quenching of the obtained emulsion leads to obtain solid particles having SPAN of 1,0 and an average size of 68  $\mu\text{m}$ . Therefore, solid particles characterized by a particularly narrow size distribution are obtained.

### **Example 2**

The liquid components of Example 1 are fed to the axial inlet of the first rotor-stator device with the same feeding ratio between the molten adduct and the mineral oil.

The operative conditions are such that the residence time inside each rotor-stator device is maintained at a value of 0,1 seconds, while the peripheral velocity of the rotor disks is 56,0 m/s. The  $Re_T$  inside the ducts connecting the mixing stages is of about 8400.

An emulsion comprising the molten adduct as the dispersed phase and the above mineral oil as the continuous phase is discharged from the outlet of the third mixing stage. The quenching of the obtained emulsion leads to obtain solid particles having SPAN of 0,9 and an average size of 12  $\mu\text{m}$ .

### **Example 3**

The liquid components of Example 1 are fed to the axial inlet of the first rotor-stator device at a feeding ratio (by weight) between the molten adduct and the mineral oil of 0,07.

The operative conditions are such that the residence time inside each rotor-stator device is maintained at a value of 0,1 seconds, while the peripheral velocity of the rotor disks is 56,0 m/s. The  $Re_T$  inside the ducts connecting the mixing stages is of about 8000.

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An emulsion comprising the molten adduct as the dispersed phase and the above mineral oil as the continuous phase is discharged from the outlet of the third mixing stage. The quenching of the obtained emulsion leads to obtain solid particles having SPAN of 0,8 and an average size of 9,5  $\mu\text{m}$ .

#### **Example 4 (Comparative)**

The same liquid components of Example 1 are fed to the axial inlet of a single-stage apparatus comprising only one rotor-stator device.

The feeding ratio (by weight) between the molten adduct and the mineral oil is 0,13.

The operative conditions are such that the residence time inside the rotor-stator device is maintained at a value of 0,1 seconds, while the peripheral velocity of the rotor disks is 56 m/s. An emulsion comprising the molten adduct as the dispersed phase and the above mineral oil as the continuous phase is discharged from the outlet of said rotor-stator device. The quenching of the obtained emulsion leads to obtain solid particles having SPAN of 1,4 and an average size of 16  $\mu\text{m}$ . Having a broad size distribution, the solid particles obtained in this example need to be sieved before being used in the preparation of supported catalysts for the olefin polymerization.

**CLAIMS**

1. A multistage process for the continuous production of an emulsion comprising subjecting at least two immiscible liquids to a sequence of at least two mixing stages carried out in at least two successive stator-rotor devices, wherein a peripheral outlet from a first stator-rotor device is connected to an axial inlet in the successive stator-rotor device by means of a duct in which the Reynold number  $Re_T$  inside said duct is higher than 5000, and the peripheral velocity of each rotor of said stator-rotor devices ranges from 5 to 60 m/s.
  2. The process according to claim 1, wherein the dispersed phase of said emulsion comprises a molten adduct of magnesium dihalide-Lewis base.
  3. The process according to claims 1-2, wherein the continuous phase of said emulsion is a liquid which is inert and immiscible with said molten adduct of magnesium dihalide-Lewis base.
  4. The process according to claim 3, wherein said liquid is selected from aliphatic and aromatic hydrocarbons, silicone oils, liquid polymers or mixtures of said compounds.
  5. The process according to claims 1-4, wherein said molten adduct of magnesium dihalide-Lewis base is fed to the first mixing stage with a weight ratio of less than 0,5 with respect to said liquid.
  6. The process according to claims 1-5, wherein in each mixing stage the residence time is of less than 1 second.
  7. The process according to claims 1-6, wherein the peripheral velocity of each rotor disk is comprised in the range from 20 to 60 m/sec.
  8. The process according to claims 1-7 wherein  $Re_T$  inside said duct is higher than 8000.
  9. Process according to claims 1-8 comprising a sequence of three mixing stages.
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10. Process according to claim 2, wherein the magnesium halide is magnesium chloride.
  11. Process according to claim 2, wherein the Lewis base is selected from amines, alcohols, esters, phenols, ethers, polyethers, aromatic or aliphatic (poly)carboxylic acids.
  12. Process according to claim 11, wherein the Lewis base is an alcohol of formula ROH in which R is an alkyl group containing from 1 to 10 carbon atoms.
  13. Process according to claims 2 and 10-12, wherein said molten adduct is  $\text{MgCl}_2 \cdot m\text{ROH} \cdot n\text{H}_2\text{O}$  in which m ranges from 0,1 to 6, n ranges from 0 to 0,7 and R is an alkyl group containing from 1 to 10 carbon atoms.
  14. Process according to claim 13 wherein m ranges from 2 to 4, n ranges from 0 to 0.4 and R is ethyl.
  15. An apparatus for the continuous production of an emulsion comprising a plurality of at least two stator-rotor devices, each stator except the last being connected with the successive stator by a duct extending from a peripheral outlet in the first stator to an axial inlet in the successive stator.
  16. The apparatus according to claim 15, wherein the initial portion of said duct is oriented in a direction substantially tangential to the circumference of each rotor.
  17. The apparatus according to claim 15, wherein the end portion of said duct is oriented in a direction substantially parallel to the rotation axes of each rotor.
  18. The apparatus according to claims 15-17, wherein said duct is shaped as a spiral.
  19. The apparatus according to claims 15-18, wherein each rotor is perforated by one or more holes allowing the emulsion to pass from one side to the other side of said rotor.
  20. The apparatus according to claims 15-19, wherein the axial tolerance between each rotor and the corresponding stator is from 0,1 to 2 mm, preferably from 0,2 to 1,2 mm.
  21. The apparatus according to claims 15-20, wherein the radial tolerance between the
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circumference of each rotor and the corresponding stator is from 0,2 to 5,0 mm, preferably from 0,5 to 2,0 mm.

**ABSTRACT**

A multistage process for the continuous production of an emulsion comprising subjecting at least two immiscible liquids to a sequence of at least two mixing stages carried out in at least two successive stator-rotor devices, wherein a peripheral outlet from a first stator-rotor device is connected to an axial inlet in the successive stator-rotor device by means of a duct in which the Reynold number  $Re_T$  inside said duct is higher than 5000, and the peripheral velocity of each rotor of said stator-rotor devices ranges from 5 to 60 m/s.

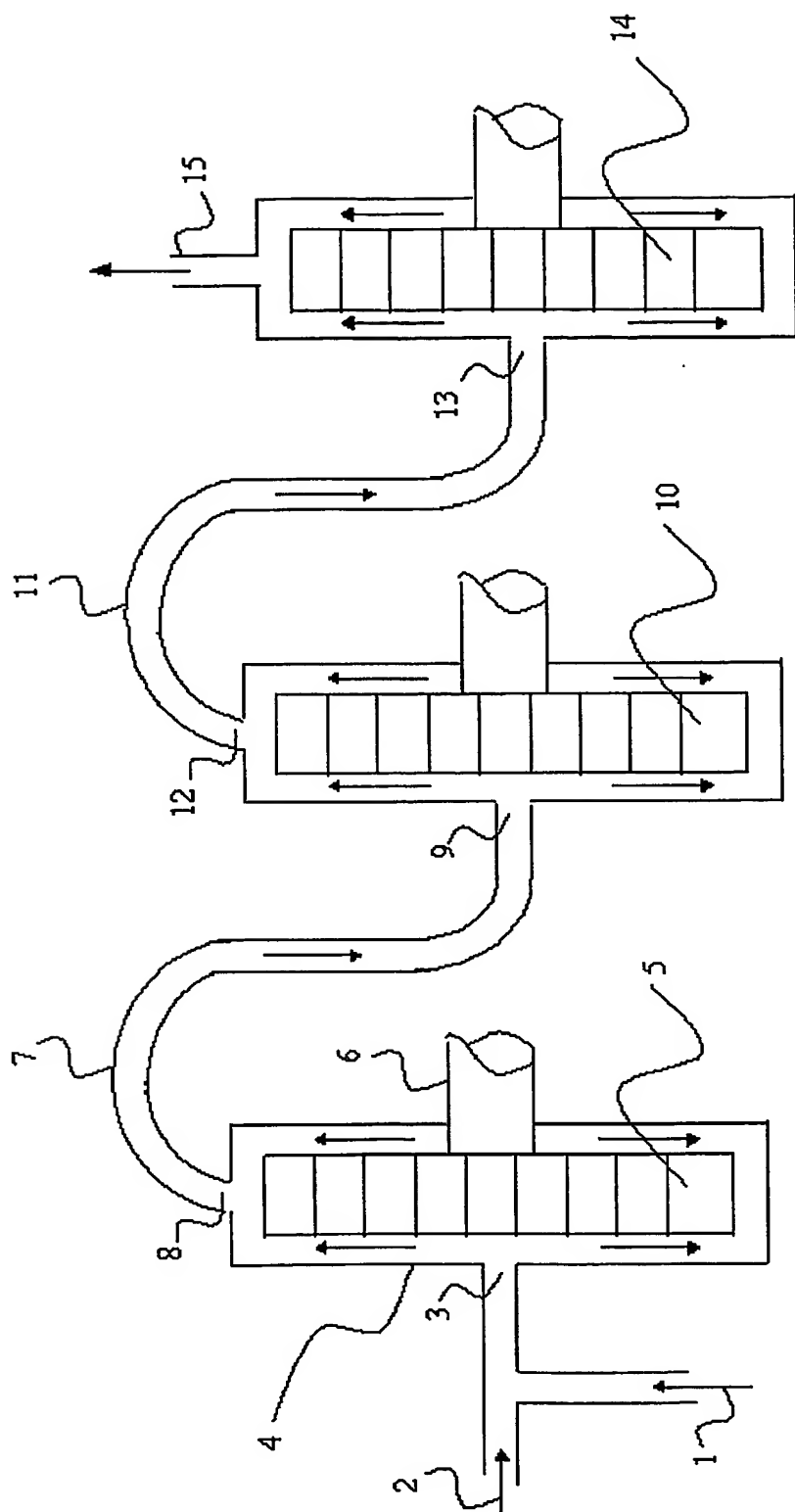


FIG. 1

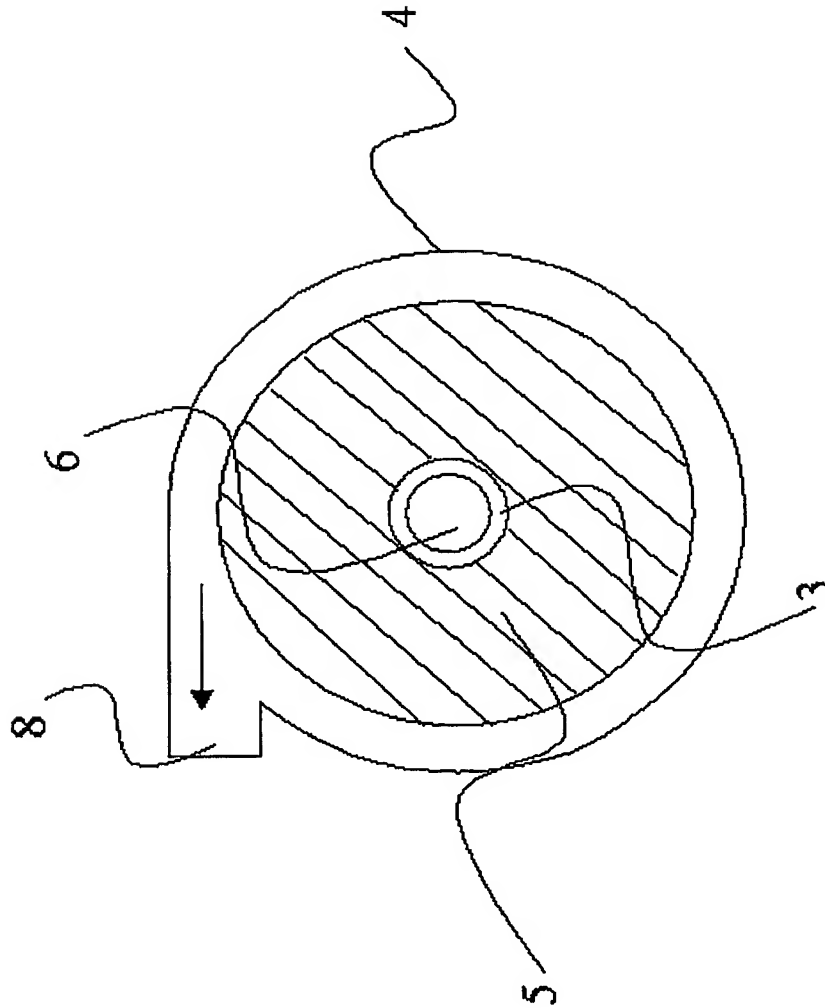


FIG. 2



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